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(54) Title: ANT SPRAY CONTAINING D-LIMONENE AND METHODS OF MAKING AND USING SAME (57) Abstract An insecticidal composition effective in controlling insects, such as ants, aphids, mealy bugs, white flies, spider mites, leaf hoppers, cabbage loopers, leaf eating beetles and caterpillars, cockroaches, flies, wasps, body and head lice but which is non-toxic to humans and household animals comprising between about 1 % to about 20 % by weight of D-limonene, between about 1 % to about 25% by weight of a non-toxic emulsifying agent, between about 0.01 % to about 5 % by weight of a preservative, and the balance of water.		

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ANT SPRAY CONTAINING D-LIMONENE AND METHODS OF MAKING AND USING SAME

Field of Invention

The present invention generally relates to an insecticidal composition that is effective in controlling insects including ants, aphids, mealy bugs, white flies, spider mites, leaf hoppers, cabbage loopers, leaf eating beetles and caterpillars, cockroaches, flies, wasps, body and head lice and more particularly relates to an insecticidal composition that contains D-limonene, a non-toxic hydrophilic solvent, a non-toxic emulsifying agent, and a preservative, that is effective in repelling and killing insects but is non-toxic to humans and household animals, and not harmful to landscaping, particularly rose bushes and ornamentals, indoor plants or the environment.

Background of the Invention

Numerous pesticide and insecticide products are available in the market for killing insects such as ants. However, these products are generally toxic to humans and household animals, and harmful to plants and the environment. Therefore they must be administered with extreme care. This is especially a problem in the termination of ants, cockroaches and the like because they normally appear near food where a careless use of insecticides may cause accidental poisoning of humans and household animals.

Other insecticide products containing D-Limonene disperse the D-Limonene in mineral oil petroleum distillates which may be harmful if accidentally ingested and may cause damage to plants. Unlike the compositions of the present invention, no emulsifier is used because the D-Limonene is dispersed in oil and not water.

Lice infestation of humans, particularly children, is still treated with compositions that contain the pesticide lindane. Lindane may be carcinogenic. There exists a need for a composition which can treat body and head lice but which is safe to use, particularly on children.

U.S. Patent No. 4,379,168 to Dotolo discloses pesticides containing D-limonene as an insect-killing ingredient along with water-soluble surfactants or emulsifiers, and water. The pesticide compositions are designed for use mainly as a dip to rid small animals of fleas and ticks and as a spray to kill fleas and ticks on small animals. None of the compositions taught by Dotolo contain suitable amounts of emulsifying agent and D-limonene for the purpose of the present invention. None of the compositions taught by Dotolo contain any surfactants or emulsifiers that were selected for their safety. For example, the Kodak Laboratory Chemical Catalog No. 51 indicates that Triton X-100, which is disclosed as an acceptable emulsifier in Dotolo, is irritating to the skin and eyes.

U.S. Patent No. 3,023,144 to Greathouse, et al., discloses germicides and fungicides containing about 25% by weight D-limonene, about 1% by weight of p-methyl acetophenone, and other unsaturated hydrocarbon cleavage products of D-limonene, up to about 7% by weight concentrated citrus oil foots and from 0.25% up to about 10% by weight salicylic acid. The compositions are used for topical application on humans and animals to control infections of skin and external organs arising from wounds or from infestation by fungi, bacteria, and larvae. Greathouse discloses that the active ingredient for the biocidal activity of the compositions disclosed is not D-limonene but rather compounds such as p-methyl acetophenone, and other unsaturated hydrocarbon cleavage products of D-limonene.

It is therefore an object of the present invention to overcome the various drawbacks associated with the use of prior art pesticide compositions.

It is another object of the present invention to provide an insecticidal composition that controls insects such as ants, aphids, mealy bugs, white flies, spider mites, leaf hoppers, cabbage loopers, leaf eating beetles and caterpillars, cockroaches, flies, wasps, body and head lice by repelling them, and which is non-toxic to humans, household animals and house plants.

It is yet another object of the present invention to provide an insecticidal composition that is effective in controlling insects such as ants, aphids, mealy bugs, white flies, spider mites, leaf hoppers, cabbage loopers, leaf eating beetles and caterpillars, cockroaches, flies, wasps, body and head lice by killing them, and which is non-toxic to humans, household animals and house plants.

It is another object of the present invention to provide a safe and effective treatment for lice infestation of a human.

It is another further object of the present invention to provide an insecticidal composition that contains an insecticide made from a natural substance that is part of and therefore not harmful to landscaping, particularly rose bushes and ornamentals, indoor plants or the environment.

It is yet another further object of the present invention to provide an insecticidal composition that contains D-limonene, a non-toxic emulsifying agent, a preservative, and a non-toxic hydrophilic solvent.

It is yet another further object of the present invention to provide an insecticidal composition that contains D-limonene, a non-toxic emulsifying agent, a preservative, and water and is suitable for application as a spray.

Summary of the Invention

In accordance with the present invention, insecticidal compositions and methods of making and of using such compositions are disclosed.

A new and novel insecticidal composition can be made by use of the naturally occurring substance of D-limonene as a major ingredient of the insecticide. D-limonene can be obtained from steam extraction of citrus peels of orange, lemon, lime, grapefruit and bergamot, some of the extractions can contain as high as 90% D-limonene. The process therefore provides a valuable use of what would otherwise be a waste product. Distillation of the oils produces technical grades of D-limonene of higher purity, *i.e.*, from about 95% to about 96%. D-limonene has a pleasant citrus scent. It can be suitably used in any living environment.

An insecticidal composition according to the present invention can be formulated by using an amount of D-limonene sufficient to provide insect control, a non-toxic hydrophilic solvent, an amount of a non-toxic emulsifying agent sufficient to solubilize the D-limonene in the non-toxic hydrophilic solvent.

An insecticidal composition according to the present invention can be formulated by using between about 1% to about 20% by weight of D-limonene, between about 1% to about 25% by weight of a non-toxic emulsifying agent, such as Alkamuls EL620, between about 0.01% to about 5% by weight of a preservative, and the balance of a non-toxic hydrophilic solvent.

A preferred concentration range is between 1% to about 10% by weight of D-limonene, between about 5% to about 15% by weight of a non-toxic emulsifying agent, such as Alkamuls EL620, between about 0.01% to about 1% by weight of a preservative, and the balance of water.

The present invention further teaches a method of using an insecticidal composition by applying such composition by, for example, spraying either directly on insects, such as ants, or in areas where insects, such as ants, frequently appear. The non-toxic insecticidal composition has a pleasant citrus scent and is suitable for use in any living environment. It was further discovered that when the present invention is applied to solid surfaces and left to remain there, the residual effect of the insecticidal composition will last for a period of time effectively keeping insects, such as ants, away from the treated area.

The present invention also teaches a method of applying the present insecticidal composition on a rose bush or other ornamental plant to control insects.

The present invention also teaches a method of using an insecticidal composition to treat humans, particularly children, infected with lice, particularly head lice.

Detailed Description of Preferred Embodiment

The present invention can be formulated by utilizing the naturally occurring substance D-limonene. D-limonene, otherwise known as orange limonene or 1-methyl-4-(1 methylethenyl) cyclohexene or 4-isopropenyl-1-methyl cyclohexene has a chemical formula of $C_{10}H_{16}$, a molecular weight of 136.2, and contains 88.1% C and 11.8% H by weight. It occurs in various ethereal oils, particularly in oils of lemon, orange, lime, grapefruit and bergamot. The D-form of limonene is a liquid having a boiling point of 175.5-176 degrees centigrade. It can be commercially obtained from Lykes Pasco Packing Company (Dade City, Florida) or Florida Chemical Company (Lake Alfred, Florida).

The compositions of the present invention allow a user to provide insect control in interior and exterior settings. Insect control can include repelling and/or killing insects, such that less insects are alive or present in a given area than if the compositions of the present invention had not been applied in the area.

While not limiting the invention by any particular theory, it is believed that the D-limonene acts to soften the waxy coating on the exoskeleton of insects and thereby causes the softened coating to clog the external insect respiratory organs, known as spiracles. The clogged spiracles interfere with the ability of the insect to obtain adequate amounts of oxygen, ultimately resulting in the death of the insect. This invention is therefore also suitable against other pests, besides insects, which would be susceptible to the external effects set forth above.

An emulsifying agent is necessary to disperse the D-limonene evenly in a non-toxic hydrophilic solvent, such as water. Other non-toxic hydrophilic solvents, for example, ethanol, dilute acetic acid solutions, and the like can be suitably used. It was discovered that a suitable emulsifying agent should be a non-toxic type such as a polyethoxylated castor oil. One such emulsifying agent is available commercially under the trade name of Alkamuls EL620 from Rhone Poulenc Co. It is non-toxic to humans, household animals and house plants and landscaping and will not cause skin or eye irritation. Other commercially available emulsifying agents that are non-toxic such as polyoxyethylenesorbitans supplied by ICI Americas or Sigma Chemical Company may also be suitably used for the present invention. In a preferred embodiment a polyoxyethylenesorbitan monooleate such as Tween 80 may be used.

In general, the emulsifying agent should be present in an amount sufficient to render the D-limonene soluble in the non-toxic hydrophilic solvent. When a polyethoxylated castor oil is used, it should contain sufficient polyethoxylation to render the D-limonene soluble in a non-toxic hydrophilic solvent when the emulsifying agent is used in an amount as disclosed herein.

In order to provide a reasonable shelf-life to the insecticidal compositions, it is preferable that a preservative be added to the composition. One such suitable preservative is

sodium benzoate commercially supplied by Pfizer, Inc. Other commercially available preservatives used for preserving food, as would be known to those of ordinary skill in the art, may also be suitably used.

A novel insecticidal composition which may be suitable for application as a spray can be formulated with between about 1% to about 20% by weight of D-limonene, between about 1% to about 25% by weight of Alkamuls EL620 (a non-toxic emulsifying agent), between about 0.01% to about 5% by weight of a preservative, and the balance of water. A more preferred composition contains between about 1% to about 10% by weight of D-limonene, between about 5% to about 15% by weight of Alkamuls EL620, between about 0.01% to about 1.0% by weight of a preservative, and the balance of water.

A specific example of the present invention is made of about 5.8% by weight of D-limonene, about 10% by weight of Alkamuls EL620 (polyethoxylated castor oil), about 0.1% by weight of sodium benzoate, and the balance of water.

The insecticidal composition made by the above specific formulation has a white, opalescent color. Its physical state is a liquid at 25 degrees Centigrade and has a citrus-like odor. It has a boiling point between 99-100 degrees Centigrade and a specific gravity of 0.9753 gm/ml. It is soluble in water and has a pH of 6.1. The flash point of the insecticidal composition is greater than 60 degrees Centigrade. It has a viscosity of 1.97 centipoise at 37.8 degrees Centigrade.

The insecticidal composition formulated according to the present invention is non-toxic to humans and household animals such as dogs, cats, rabbits, etc. It can be used near food without any danger of contamination or accidental poisoning. It will not harm landscaping foliage or indoor plants. It leaves a pleasant citrus-like smell which is not objectionable to most people. Since it is made of mainly naturally occurring material, it is not

harmful to the environment and does not cause any unwanted pollution. It is also completely biodegradable.

When the novel insecticidal composition is used indoors as a spray, it should be sprayed preferably from about 6 to about 8 inches away from and directly on insects such as ants, or on insect trails such as ant trails to the source of the insects such as ants and sprayed until visibly wet. Insects are typically killed within minutes of contact with the novel insecticidal compositions.

The insecticidal composition can be used on the interior surfaces in a building such as counter tops and in food preparation areas. For outdoor use, the insecticidal composition of the present invention should be applied at the perimeter of a building such as a home, at insect trails such as ant trails, at insect nests, such as ant nests and at doors, cracks, and window frames where insects, such as ants may enter the building. It should be applied until visibly wet. It will provide lasting repellent qualities.

Application of the present insecticidal compositions is preferably effected by spraying of the insecticidal compositions by conventional spray apparatus such as aerosol cans bug sprayers and the like. However, application may also be effected by any means of contacting surfaces to be treated, for example, with a brush which has been dipped in the insecticidal compositions. Because the novel insecticidal compositions are non-toxic, they can also be applied with a human hand.

When the novel insecticidal composition is used to treat humans infected with lice, it should be applied to the infected area, such as the scalp and left on for about 5 minutes after which it may be rinsed and preferably shampooed off.

While this invention has been described in an illustrative manner, it should be understood that the terminology used is intended to be in the nature of words of description

rather than of limitation. Furthermore, while the invention has been described in terms of a preferred embodiment thereof, it is to be appreciated that those skilled in the art will rapidly apply these teachings to other possible variations of the invention.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

What is claimed is:

1. An insecticidal composition comprising an amount of D-limonene sufficient to provide insect control, a non-toxic hydrophilic solvent, an amount of a non-toxic emulsifying agent sufficient to solubilize said D-limonene in said non-toxic hydrophilic solvent, wherein said spray is non-toxic to human and household animals.
2. The insecticidal composition according to claim 1 wherein said amount of D-limonene is between about 1% to about 20% by weight, said amount of non-toxic emulsifying agent is between about 1% to about 25% by weight, and said non-toxic hydrophilic solvent is provided in an amount between about 98% to about 55% by weight.
3. The insecticidal composition according to claim 1 further comprising an amount of preservative sufficient to extend the effectiveness of said insecticidal composition.
4. The insecticidal composition according to claim 3 wherein said amount of preservative is between about 0.01% to about 5% by weight.
5. The insecticidal composition according to claim 1 wherein said amount of D-limonene sufficient to provide insect control is sufficient to repel an insect.
6. The insecticidal composition according to claim 1 wherein said amount of D-limonene sufficient to provide insect control is sufficient to kill an insect.
7. The insecticidal composition according to claim 1 wherein said non-toxic emulsifying agent is a polyethoxylated castor oil.
8. The insecticidal composition according to claim 1 wherein said non-toxic emulsifying agent is a polyoxyethylenesorbitan.
9. The insecticidal composition according to claim 1 wherein said non-toxic hydrophilic solvent is water.

10. The insecticidal composition according to claim 2 wherein said preservative is a food preservative.

11. The insecticidal composition according to claim 1 wherein said insects are selected from the group consisting of ants, aphids, mealy bugs, white flies, spider mites, leaf hoppers, cabbage loopers, leaf eating beetles and caterpillars, cockroaches, flies, wasps, body lice and head lice.

12. The insecticidal composition according to claim 1 wherein said insecticidal composition preferably comprises between about 1% to about 10% by weight of D-limonene, between about 5% to about 15% by weight of a non-toxic emulsifying agent and between about 94% to about 75% by weight of a non-toxic hydrophilic solvent.

13. The insecticidal composition according to claim 2 comprising about 5.8% by weight of D-limonene, about 10% by weight of polyethoxylated castor oil, about 0.1% by weight of sodium benzoate, and about 84.1% by weight of water.

14. An insecticidal composition produced by a method comprising the step of combining between about 1% to about 20% by weight of D-limonene, between about 1% to about 25% by weight of a non-toxic emulsifying agent, between about 0.01% to about 5% by weight of a preservative, and between about 97.99% to about 50% by weight of water.

15. A method of controlling insects comprising the step of applying the insecticidal composition of claim 1 to the group consisting of an insect, an insect trail, a source of an insect, an interior surface of a building, an outside perimeter of a building or combinations thereof.

16. The method of controlling insects according to claim 15 wherein said insecticidal composition preferably comprises between about 1% to about 10% by weight D-limonene, between about 5% to about 15% by weight of a non-toxic emulsifying agent, between about 0.01% to about 1% by weight of a preservative and between about 93.99% to about 74% by weight of a non-toxic hydrophilic solvent.

17. A method of treating a human infected with lice comprising the steps of applying the composition of claim 1 to a portion of the human infected with the lice, allowing said composition to remain on the infected portion for a period of time and removing said composition.

18. The method of claim 17 wherein said insecticidal composition preferably comprises between about 1% to about 10% by weight of D-limonene, between about 5% to about 15% by weight of a non-toxic emulsifying agent, between about 0.01% to about 1% by weight of a preservative, and between about 93.99% to about 74% by weight of a non-toxic hydrophilic solvent.

19. The method of claim 17 wherein said insecticidal composition preferably comprises between about 5.8% by weight of D-limonene, about 10% by weight of polyethoxylated castor oil, about 0.1% by weight of sodium benzoate, and about 84.1% by weight of water.

20. A method of controlling insects on plants, said plants selected from the group consisting of rose bushes and ornamentals comprising the step of applying the composition of claim 1 to said plants.

INTERNATIONAL SEARCH REPORT

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PCT/US98/08954

A. CLASSIFICATION OF SUBJECT MATTER

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US CL : 514/763

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 514/763

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 3,930,010 A (KLOPPING) 30 December 1975, see entire document.	1-20
Y	US 5,194,264 A (VAN TONDER) 16 March 1993, see entire document.	1-20
Y	US 4,379,168 A (DOTOLO) 05 April 1983, see entire document.	1-20

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

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